Simplified Wire-Screen Packing for Fractionating Columns

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An efficient, inexpensive wire-screen packing is described which is easy to fabricate. It requires no welding, grinding, precise fitting, or preassembly of parts, and ordinary glass or metal tubing is sufficiently uniform in bore for use with the packing. Fractionating efficiency, liquid holdup, and pressure drop are given for three columns 0.625, 1, and 2 inches in inside diameter. These operating characteristics compare favorably with those of other types of screen-packed columns, which require much more skill, precision, and expense in fabrication.

In recent years, several efficient laboratory fractionating columns having wire-screen packing have been described (*1–4*). Although these columns have high efficiency (as indicated by low height equivalent to a theoretical plate), their widespread use in routine work is severely limited by their high cost, which is due to the skill and precision required in their construction.

This paper describes a form of wire-screen packing which is inexpensive to fabricate and yet is highly efficient in use. The individual screen elements are not welded or otherwise united; this makes possible the use of metal screen which would be difficult or impossible to spot weld. Furthermore, because the screen elements are individually packed into the column without any preassembly, and as placed in the column, they are under stress, they expand to fit the tube snugly despite variations in tube diameter and deviations from a true circular cross section. This prevents leakage of liquid past the elements and permits the use of ordinary tube or pipe instead of precision-bore tube.

CONSTRUCTION OF THE COLUMN

Heavy-walled Pyrex tubing or Pyrex pipe was used in most cases because of the internal pressure on the tube produced by the stress in the screen elements and retaining rings.

The wire screen was stainless steel, Phosphor bronze, brass, or copper, the last two being less satisfactory because of their poor elasticity and tendency to relax under mild heat. For maximum efficiency the screen elements should maintain close contact with the column wall by virtue of firm and unrelaxing pressure owing to the elasticity of the metal. No study of mesh size as related to

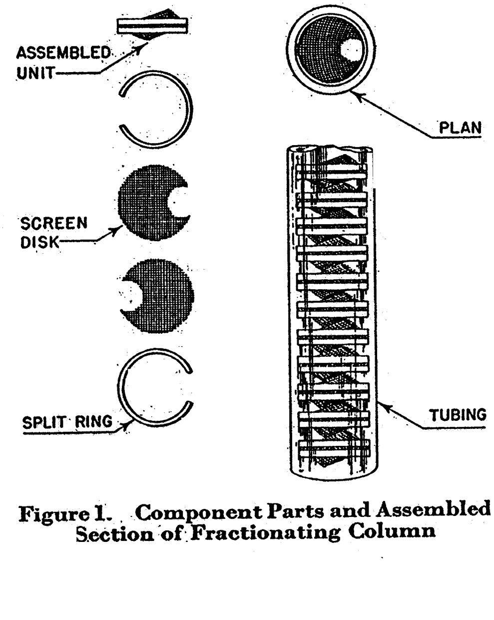

Figure 1. Component Parts and Assembled Section of Fractionating Column

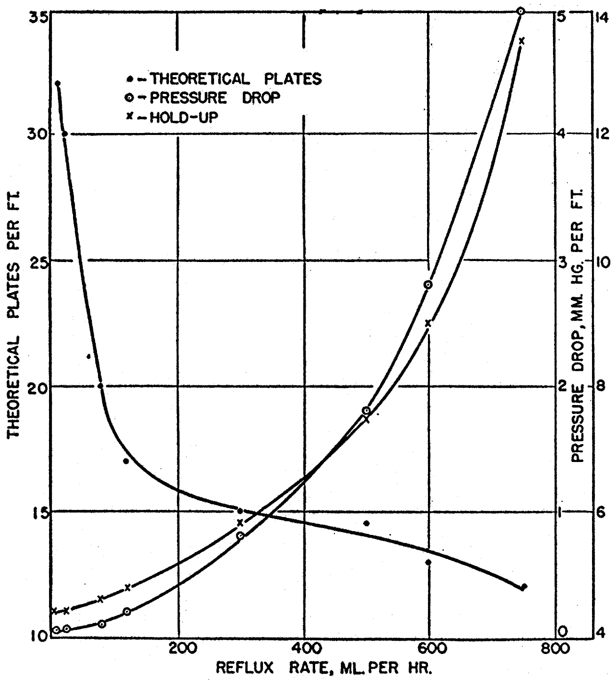

Figure 2. Performance of Column 1

efficiency was made, but this is thought to be relatively unimportant as long as the mesh is fine enough for the liquid to fill the interstices, thus forming a continuous film over the surface of the screen. The screen used in the present work was 60 by 60 or 80 by 80 mesh.

Figure 1 shows the component parts and an assembled section of packing. Circular screen disks are cut out with a punch press. Then a portion of a circle is cut from the edge of each disk to a depth of about one third the diameter of the disk. This portion which was cut out serves as the vapor passage in the assembled packing and may be varied in size. Increasing the radius, or depth, of the cut increases the size of the vapor passage, thus increasing the capacity of the column. At the same time, however, it reduces the area of screen surface and hence slightly reduces the efficiency of the packing.

The diameter of the screen disks should be 5 to 10% greater than the diameter of the tube in which they are assembled. Then, when the disk is forced into the tube, it will be bowed and will remain under stress, because of the elasticity of the metal. This stress serves the double purpose of holding the disk snugly in place and making it shape itself closely to the wall of the tube. Thus a tight fit is obtained despite variations in the diameter of the tube or departures from a true circular cross section in the tube. This close contact between the edges of the disks and the wall of the tube is extremely important; otherwise liquid reflux would leak through and channel down the side of the tube.

The assembled unit, or cell, shown in Figure 1 consists of two disks bowed in opposite directions (with their concave sides together) and having their vapor openings on opposite sides of the column. In order to be doubly sure that a tight seal is obtained and that the cells are held firmly in place, split rings similar to piston rings are placed above and below each cell. These rings are made of stainless steel, Phosphor bronze, or other elastic material which retains its elasticity at the temperatures which will be encountered in the use of the column. The assembled packing consists of a series of such cells placed so that each touches those above and below, thus providing a continuous screen surface over which the reflux flows and a zig-zag open pathway up through which the vapor passes.

ASSEMBLY OF STILLS

For determination of operating characteristics, the columns were enclosed in double glass jackets, the inner jacket having a single winding of Nichrome ribbon. Thermometers were placed near the top and the bottom of the columns inside the inner jacket. Heat input, controlled by a Variac, was adjusted so that the thermometers near the top of the jacket read the same as the one in the vapor at the still head. The still pot was a round-bottomed flask heated with a Glas-Col heater controlled by a Variac. The

Table I. Specifications of Columns

Column No.	1	2	3
Type tube	Pyrex combustion	Pyrex pipe	Pyrex medium wall
Inside diameter, mm.	17	25.4	50.8
Packed length, inches	12	9.5	11
Number of screen disks	76	48	22
Diameter of disks, mm.	19	28.6	57.2
Metal used	Phosphor bronze	Stainless steel	Phosphor bronze
Mesh of screen	60 × 60	80 × 80	60 × 60
Diameter wire, inches	0.0075	0.0055	0.0075
Size stock for rings, inches	$^1/_{16} \times ^1/_{32}$	$^1/_{16} \times ^1/_{32}$	$^1/_8 \times ^1/_{16}$

pot had a side inlet with a 10/30 ground-glass joint through which a tube extended. This inlet tube was used to withdraw samples for analysis and also for connection to a manometer for measurement of pressure drop through the column. The still head used was sometimes of the Rossini type (*6*) and sometimes of a simpler total condensation variable take-off type having a holdup of 0.2 to 0.5 ml.

All connections between pot, column still head, and thermometer were standard taper ground-glass joints.

DETERMINATION OF OPERATING CHARACTERISTICS

Of the numerous co umns constructed and evaluated in one or more respects, three were chosen for more complete testing because their size and design best fitted them for general use in the usual types of laboratory fractionating problems. Table I shows the pertinent data on their construction and Figures 2, 3, and 4 show their operating characteristics. The test mixture used was *n*-heptane and methylcyclohexane, and analyses were made by refractive index, the data quoted by Ward (*5*) being used. Efficiency tests were all made under total reflux. Pressure drop through the columns was measured by a U-tube manometer con-

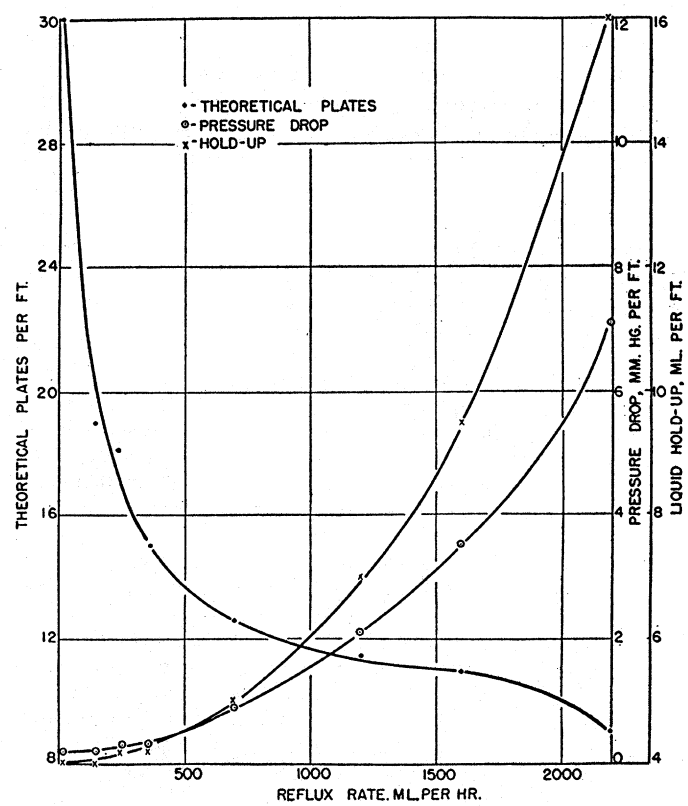

Figure 3. Performance of Column 2

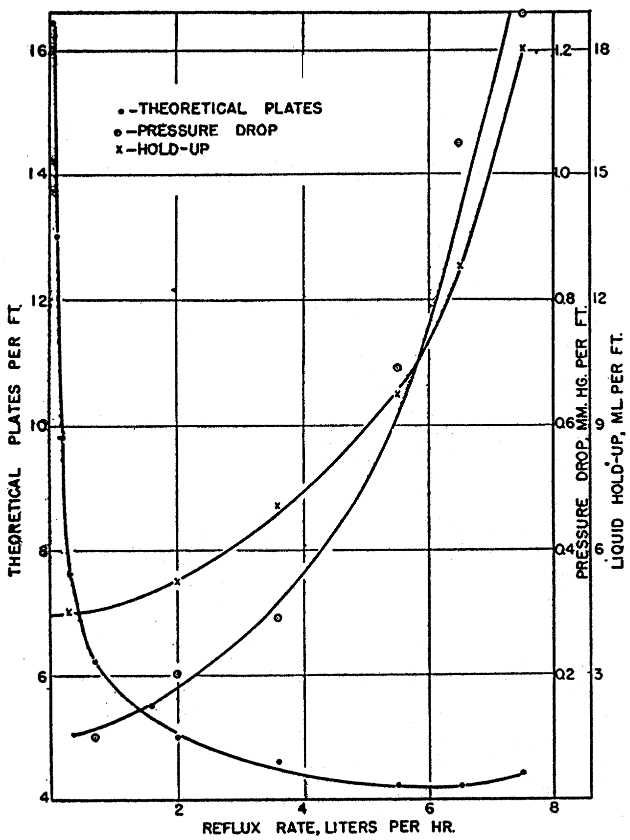

Figure 4. Performance of Column 3

nected to the still pot and open to the atmosphere on the other arm. Holdup was measured by the method recommended by Ward; heptane and biphenyl ether were used and analyses were made by refractive index.

The rate of boilup and the capacity of the columns were determined by operating with total take-off for a brief measured interval (0.5 or 1.0 minute) and measuring the volume of distillate. Thus, the rates were measured at the still head and not at the pot, though there should be little difference in rates at the two points.

No tests were run beyond the point of incipient flooding because of the difficulty of maintaining steady conditions for a period sufficient to give reliable and reproducible results. The maximum rate of boilup that could be obtained from a 5-liter flask heated with a Glas-Col heater was 7500 ml. per hour. This was not sufficient to flood Column 3 (Table I and Figure 4); hence the characteristics of this column are shown in Figure 4 only up to this boilup rate and not to its flood point. The behavior of Column 3 at the boilup rate of 7500 ml. per hour indicated that its maximum capacity was at least 8 or 9 liters per hour.

Any attempt to extrapolate the operating characteristics as shown in Figures 2 to 4 to columns of other diameters is complicated by several factors: The number of disks per foot of column was not constant; the percentage of the area of the disks cut out to form the vapor path was not exactly constant; and the ratio of the diameter of the disks to that of the tubing was not constant in the three columns described. However, a few qualitative statements about the effect of increasing the diameter on operating characteristics seem justified. The number of theoretical plates per foot is markedly reduced at larger diameters, both because of fewer disks per foot being used and because of increasing difficulty of maintaining close contact between disks and tubing. Pressure drop per foot of column decreases, primarily because of the presence of fewer disks per foot. The liquid holdup increases somewhat. The greater area of wet surfaces accounts for this, but the effect is diminished by the presence of fewer disks per foot.

Like other types of screen-packed columns, it is essential that the packing be thoroughly and uniformly wet before beginning a distillation or test run. This is accomplished easily by a short period of rapid boilup under total reflux. When wet, a uniform continuous film of liquid covers all screen surfaces. This film remains intact so long as reflux is continued, no matter how low the boilup rate. Maximum plate efficiency was obtained at very low boilup rates (barely enough to keep the packing wet).

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